

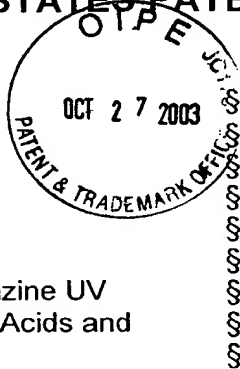
IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Application of: Gupta *et al.*

Application No.: 09/779,597

Filed: February 9, 2001

For: Process for Making Triazine UV
Absorbers Using Lewis Acids and
Reaction Promoters



Art Unit: 1624

Examiner: V. Balasubramanian

Docket No.: 98064-02

Date: July 11, 2003

DECLARATION UNDER 37 C.F.R. Section 1.131(a)

We, Ram B. Gupta, Dennis J. Jakiela, Sampath Venimadhavan, Russell C. Cappadona and Venkatrao K. Pai, being duly sworn depose and say:

That we are the inventors for the above-identified patent application;

That we conceived and reduced to practice in the United States the invention claimed in the above-identified patent application prior to March 2, 1998, the priority filing date of the cited U.S. Patent No. 6,242,598 B1 to Stevenson et al.

Attached Exhibit A is a copy of notebook records relating to the reduction to practice of our invention, wherein an aryl substituted 1,3,5-triazine forming reaction facilitator comprising a Lewis Acid ($AlCl_3$), a reaction promoter (conc. HCl-protic acid) and a solvent (chlorobenzene) is used to prepare aryl substituted 1,3,5-triazine compounds CDMP (2-chloro-4,6-bis(2,4-dimethylphenyl)-1,3,5-triazine) and P-1062 (2-(2,4-dihydroxyphenyl)-4,6-bis(2,4-dimethylphenyl)-1,3,5-triazine).

That Exhibit A, which relates to the aforementioned actual reduction to practice, corresponds to the invention broadly disclosed and claimed in the above-identified patent application.

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

/s/ Ram B. Gupta July 11, 2003
(Ram B. Gupta)

/s/ Dennis J. Jakiela July 11, 2003
(Dennis J. Jakiela)

/s/ S. Venimadhavan July 15, 2003
(Sampath Venimadhavan)

/s/ Russell C. Cappadona July 11, 2003
(Russell C. Cappadona)

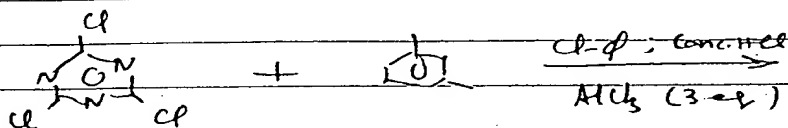
/s/ Venkatrao K. Pai July 11, 2003
(Venkatrao K. Pai)

PROBLEM NO.

CONTINUED FROM:

PURPOSE OF THE PROCEDURE

Reaction of cyanuric chloride with m-xylene
containing 3.0 eq. AlCl₃ and 0.4% conc. HCl in chlorobenzene



| | <u>FW</u> | <u>Am't. Used</u> | <u>MMs</u> |
|--------------------|-----------|-------------------|------------|
| cyanuric chloride | 184 | 3.68 g. | 20 mm |
| m-xylene (d=0.868) | 106 | 4.6 mL | 38 mm |
| AlCl ₃ | 134 | 8.0 gm | 60 mm |
| chlorobenzene | | 50 mL | |
| conc. HCl | | 0.4 mL | |

In a 3-neck RB flask equipped with a reflux condenser, an argon inlet, a magnetic stirring bar and glass stoppers was placed cyanuric chloride. To it was added chlorobenzene and the contents cooled in an ice bath. To it was then added AlCl₃ followed by after 5 min, conc. HCl. After 10 min. m-xylene was added. The rxn. mix. turned yellow and soon red. It was then stirred at rt. The solution has become dark red. A sample was withdrawn & analyzed after 1/2 hr. by HPLC. The analysis showed 97% cc conversion to mainly COMPT & triethyl formed in 96:4 ratio. The reaction mix. was stirred for additional 1/2 hr & a sample again analyzed by HPLC. It showed > 98% conversion of CC to COMPT & triethyl formed in 95:5 ratio. The reaction mixture was left overnight stirring at rt.

A final sample was analyzed after 20 hr at room temp. by HPLC. It showed 99.5% conversion of cc to only two products, viz., COMPT & triethyl formed in 96:4 ratio. This is the best ratio we have observed thus far.

SIGNED

RB Smith

DATE:

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S. Law

DATE:

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PURPOSE OF THE PROCEDURE

Further Reaction with Resorcinol

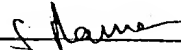
To the stirring reaction mixture at room temp. was added 2.42 gm (22 mm) of resorcinol. It was then heated to 85°C and held at this temp. for 1.5 hr. The heating was discontinued at this stage and the reaction mixture left stirring at room temp. overnight.

A sample was withdrawn and analyzed by HPLC. It showed 92.7% P-1062, 4.99% Bisoxyl and 2.25% Unreacted CMPT. After complete conversion of CMPT the ratio of P-1062 to Bisoxyl can be as high as 95.7% : 4.3%.

SIGNED



DATE:

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DATE:

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